

## **MEASUREMENT OF CAPILLARY PRESSURE CURVES AT RESERVOIR CONDITIONS**

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### **ABSTRACT**

This paper presents the first measurement of a full cycle of capillary pressure curves at reservoir conditions (300 bar or 4,300 psi and 130°C). The measurement is based on the semi-dynamic method already described in previous SCA papers. The semi-dynamic method is based on a succession of steady-state displacements and is operated at reservoir conditions using a modified Hassler cell. In order to allow X-ray measurements, the core holder is made of a composite material reinforced with carbon fibers. In addition, the core holder is separately heated and a uniform temperature is achieved by circulating the confining fluid around the sample. A first experiment performed at full reservoir conditions on a carbonate sample (permeability: 13 mD - length: 6cm) is presented. Wettability is restored by aging the samples at reservoir conditions with live oil. The full cycle lasts about 2 months. No spontaneous imbibition (positive) is observed but a spontaneous displacement of brine by oil is detected (negative drainage). Capillary pressures are derived from the extrapolation of saturation profiles at the inlet of the sample. Relative permeabilities are derived by history matching of pressures and saturation profiles during the transient part of the forced displacements (negative imbibition and positive drainage). Finally, the accuracy of the method and the means to reduce the total duration of the measurement are discussed.

### **INTRODUCTION**

A SCAL (Special Core Analysis Laboratory) study is a major step in the value chain of the Integrated Reservoir Management. It provides answers to reservoir characterization and estimation of well productivity and injectivity. In this paper, we present the apparatus that has been developed at IFP to measure both capillary pressure and relative permeability under reservoir conditions at the same time and on the same plug.

The measurement technique is based on the method theoretically described by Ramakrisnan and Capiello [1], and validated at ambient conditions by Lenormand [2; 3; 4]. The advantages of the new apparatus presented in this paper are: i) the laboratory data are obtained under reservoir conditions, that can improve the representativeness of the petrophysical parameter ( $P_c$ ,  $k_r$ ); ii) the use of in-situ saturation monitoring (ISSM) improves the data interpretation.

The principle and the advantages of the semi-dynamic method are reviewed in the first part of the paper and then the new apparatus is described. The results of one experiment conducted at reservoir conditions with a carbonate sample (brine/oil fluid system) are also presented. The measured data are then interpreted in terms of capillary pressure and relative permeability and several approaches (history matching and analytical

calculations) are compared. Finally a guideline, based on numerical simulations, is proposed to optimize the  $P_c$ -measurements with the semi-dynamic method.

## **CAPILLARY PRESSURE MEASUREMENT: SEMI-DYNAMIC METHOD**

In this paper, the conventional definitions for capillary pressure are adopted (Lenormand *et al.*, 1993, [2]). The full cycle of capillary pressure is depicted in Figure 1. The different methods used to measure the capillary pressure and their principle are compared in Lenormand *et al.*, [2; 4]. The centrifuge method (O'Meara *et al.*, [5]; Forbes *et al.*, [6]) allows quick measurements of wettability with the USBM index, but is not suitable for positive (spontaneous) imbibition and negative (spontaneous) drainage. In addition, centrifuge is limited for high pressure and temperature. The porous plate method (Longeron *et al.*, [7], Fleury *et al.*, [8]) can be used to determine the whole  $P_c$ -cycle using water-wet and oil-wet semi-permeable membranes. However, the porous plate method is difficult to operate at high pore pressure and high temperature.

According to the published papers, the semi-dynamic method is the only one suitable to determine the whole  $P_c$ -cycle, for gas/oil or water/oil fluid systems, at full reservoir conditions. Furthermore, the relative permeability of the displacing phase can also be determined during the same experiment. This technique represents a very interesting fully integrated petrophysical tool for multiphase flow measurements (Lenormand, [4]).

### **Principle**

In the semi-dynamic method, the capillary pressure is balanced by the viscous pressure drop. The sample is set in a core holder, without any semi-permeable membrane. During an experiment, one fluid is injected through the sample while the second one washes the outlet face of the core. A separator is used to measure fluid production. Local saturation profiles can be measured with different techniques (ultrasonic method, conductivity, X- or  $\gamma$ -ray). A steady-state equilibrium is reached if inlet and outlet pressures and saturation profiles are both constant.

Let us consider the oil/water case, where water is the reference fluid:

- For positive capillary pressure (Figure 1), oil is injected through the sample. Water washes the outlet of the core at low flow rate,  $Q_w$ , in order to keep the water continuity in the porous medium without any disturbance of the flow in the sample. The pressure in water,  $P_w$ , is thus imposed and can be measured. If the oil flow rate,  $Q_o$ , is increased (forced drainage: curves 1 and 5 in Figure 1), water is displaced. At the steady-state equilibrium, water is no longer produced and the water pressure,  $P_w$ , is uniform along the sample (Figure 2). There is a pressure gradient for oil which is flowing. The capillary pressure at the inlet  $P_c = P_o - P_w$  is then derived from the injection oil pressure,  $P_o$ . To determine the  $P_c$ -curve, the saturation at the inlet of the porous medium is also required. This local saturation can be measured or can be

calculated. If the oil flow rate is decreased, positive imbibition is possible, thanks to the circulation of water at the outlet of the sample (curve 2 in Figure 1).

- For the negative part of the Pc-curve (Figure 1), the role of water and oil is reversed. Water is injected into the porous medium (at increasing flow rate for the negative imbibition : curve 3 in Figure 1, and at decreasing flow rate for the negative drainage : curve 4 in Figure 1) while oil washes the outlet of the sample.

For positive and negative capillary pressures close to zero, very low flow rates have to be applied in order to impose small pressure drops. The possible inlet pressure and the crossing point  $P_c=0$  cannot be investigated with this method. To determine the point  $P_c=0$  with a reasonable duration, the porous plate technique, with a very short sample, would be more suitable. With the standard centrifuge method, there is a minimum speed and the zero capillary pressure is not possible at all. However, Spinler *et al*, 1999 developed a method to determine the whole Pc-profile (positive and negative) in a sample, at a given centrifuge speed. They could thus determine quickly the point  $P_c=0$ .

### **Interpretation: Calculation of the Local Saturation**

For each injecting flow rate  $q$ , the inlet saturation  $S_i(P_i)$  and the inlet pressure  $P_i$  give a point on the Pc-curve. If local saturations are measured, the Pc-curves can be obtained directly with the collected data. If not, the inlet saturation has to be calculated analytically. Ramakrishnan and Capiello, 1991, [1] determined the inlet saturation from the average saturation  $\langle S \rangle$

$$S_i(P_i) = \langle S \rangle + q \cdot \frac{d \langle S \rangle}{dq} \quad (1)$$

and the relative permeability (Lenormand *et al*, 1997, [4]) of the injected fluid at the inlet of the sample

$$k_{r} = \frac{L \cdot \mu}{K \cdot A} \frac{dq}{dP_i} \quad (2)$$

In equation 2,  $K$  is assumed to be uniform over the sample, as for other SCAL techniques.

With only equilibrium measurements, it is thus possible to determine both capillary pressure and relative permeability.

### **Reservoir Conditions Semi-Dynamic (SDM) Device**

The semi-dynamic method was first validated at IFP at ambient conditions on sandstone and chalk outcrop samples (Lenormand *et al*. [2; 3; 4]). Since the method does not require a semi-permeable membrane, the limits in pressure and temperature are those of standard displacement experiments. For the SCAL studies, IFP developed a prototype based on the semi-dynamic method, but adapted to reservoir conditions, in order to be as representative as possible. This device can operate at 130°C and 330bar. Its principle is presented in Figure 3 and Figure 4 presents photographs of the device.

### IFP Designed Cell

The sample, 6-40cm in length and 4-5cm in diameter, is set in a particular Hassler-cell. To allow local saturation measurements, this cell is transparent to X or  $\gamma$ -ray (composite material reinforced with carbon fibers) and is equipped with an internal heating system. The confining fluid is heated with resistors set in the inlet and outlet pistons. A uniform temperature ( $\pm 1\%$ ) is achieved along the core by circulating the confining fluid around the Viton sleeve, with a rotating screw.

### Closed Loop

As for experiments at ambient conditions, the displacements are performed in a closed loop. All the elements of the loop (pistons of the four pumps, separator, valves...), except the Hassler cell and the X-ray system, are heated in an oven. Between the oven and the Hassler cell, the small lines are heated with thermal bands. During an experiment, the blind separator is used to separate the two fluids before their re-injection, and not to determine the average saturation. This average saturation is effectively very difficult to be estimated by material balance, due to the different dead volumes and to the micro-leaks that occur during a two to five months experiment. Local and average saturations are thus only measured through the X-ray system.

### X-ray System

Local saturations are measured during the experiment with the X-ray attenuation technique. The X-ray generator (90keV – Ta filter) and the detector can move along the heated Hassler cell using a step by step motor. For a better contrast, KI was used as dopant at the concentration of 30 g/l in brine. The effect of dopants on wettability was not studied. For each equilibrium, the saturation profile is calculated from the measured X-ray profile ( $N_w$ ) and the calibration profiles ( $N_w$ : sample fully saturated with brine and  $N_o$ : sample fully saturated with oil). The principle of the X-ray calculations is described in Maloney, [9]. To avoid any drift of the X-ray system, four hours of running are needed before starting the experiment. Furthermore, each profile is corrected by a reference absorption value measured on a reference material. For all the local saturation measurements, the estimated quadratic error (Norel, [10]) is between 1 and 2%.

During a Pc-cycle, one computer monitors the acquisition of X-ray data and a second one is used to control the valves and pumps and to collect pressure and flow rate data.

## **APPLICATION OF THE METHOD TO A CARBONATE SAMPLE**

### **Experiment**

The first Pc-experiments at full reservoir conditions were performed using the above described prototype. The example of a carbonate sample will be now detailed. The operating conditions were 120°C and 200bar. The petrophysical properties of the plug used are gathered in Table 1.

Table 1: Selected carbonate plug

L (cm)	d (cm)	K (mD)	$\phi$ (fraction)	Swi (fraction)
6.0	4.9	13.0	0.31	0.09

Prior to this experiment, the preserved plug was cleaned and fully saturated with reservoir brine. The irreducible water saturation was then set by the porous plate technique and the sample was aged during four weeks, at reservoir conditions, with live oil. No difference between the X-ray profiles measured before and after aging was observed.

The following Pc-cycle was then performed:

- Positive imbibition (primary imbibition : curve 2, Fig. 1) :  $Q_o=500$  and  $1\text{cm}^3/\text{hr}$
- Negative imbibition (curve 3, Fig.1):  $Q_w=1, 10, 20, 50, 200$  and  $500\text{cm}^3/\text{hr}$  –  $Q_o=20\text{cm}^3/\text{hr}$
- Negative drainage (curve 4, Fig.1):  $Q_w=500, 200, 50, 20, 10$  and  $1\text{cm}^3/\text{hr}$  –  $Q_o=20\text{cm}^3/\text{hr}$
- Positive drainage (curve 5, Fig.1):  $Q_o=1, 10, 20, 50, 200$  and  $500\text{cm}^3/\text{hr}$  –  $Q_w=20\text{cm}^3/\text{hr}$
- Positive imbibition (secondary imbibition : curve 2, Fig.1):  $Q_o=200, 50, 20, 10$  and  $1\text{cm}^3/\text{hr}$  –  $Q_w=20\text{cm}^3/\text{hr}$

For each injecting flow rate, the transient and stabilized pressures and saturation profiles ( $N_{wo}$ ) were measured. At the end of the experiment, the sample was cleaned by miscible displacements (toluene, methanol...). The calibration profiles, that is the profiles of the plug fully saturated with brine ( $N_w$ ) and fully saturated with live oil ( $N_o$ ), were then performed, at  $120^\circ\text{C}$  and  $200^\circ\text{C}$ . Finally, the saturation profiles were calculated with the profiles  $N_{wo}$ ,  $N_w$  and  $N_o$ .

## Results

Figure 5 shows the evolution of the saturation profiles with the injecting flow rate, at equilibrium, for imbibition and drainage. During the primary positive imbibition, no spontaneous displacement was observed. The saturation profiles for  $Q_o=500$  and  $Q_o=1\text{cm}^3/\text{hr}$  are the same as the  $S_{wi}$ -profile. For the forced imbibition, the water saturation increased with the water flow rate. During the spontaneous drainage, oil slightly displaced water. For the forced drainage, the water saturation decreased with the oil flow rate. No secondary spontaneous imbibition was observed (all the profiles are equivalent to the last profile of forced drainage). For each profile, the stabilized pressure was measured. The whole Pc-cycle was obtained in 2.5 months.

## INTERPRETATION OF THE SEMI-DYNAMIC EXPERIMENT

### Analytical Calculations

#### Capillary Pressure

The above experimental results were interpreted in terms of capillary pressure curves.

As the capillary pressure, that is the differential pressure, is measured at the inlet of the sample, the local saturation at the same location has to be considered. As explained above, this inlet saturation can be extrapolated from the saturation profile or can be calculated from the average saturation  $\langle S \rangle$  ( $\langle S \rangle$  is the average of the local saturations)

with the Ramakrishnan and Capiello method [1] (Equation 1). For the whole performed Pc-cycle, the second method was used. For the forced imbibition, the two methods were applied. Table 2 and Figure 6 show that, in this case, the evaluation method of the inlet saturation has little influence on the corresponding capillary pressure curve.

Table 2: Brine saturations during the forced imbibition sequence

Injection rate cm <sup>3</sup> /hr Q <sub>w</sub>	Average S <sub>w</sub> <S <sub>w</sub> >	Measured S <sub>i</sub> close to the inlet	Calculated S <sub>i</sub> Analytical calculation
1	0.508	0.558	0.560
10	0.637	0.720	0.704
20	0.673	0.783	0.755
50	0.762	0.874	0.846
200	0.843	0.913	0.897
500	0.875	0.920	0.906

The whole Pc-cycle is presented in Figure 7. The calculated USBM index (-0.07) indicates an intermediate wettability behavior. There was no spontaneous imbibition (water invasion), whereas a limited spontaneous drainage was recorded (oil invasion).

#### Relative Permeability

As underlined in the introduction, one of the advantages of the semi-dynamic method is to measure both Pc and kr-data at the same time, on the same plug. The relative permeability of the injected phase (water for forced imbibition and oil for forced drainage) at saturation S<sub>i</sub>, can be calculated straight forward knowing the injection rate and the stabilized differential pressure, using Equation 2.

The results are presented in Figure 8 for both negative imbibition and positive drainage. Even if no study on the accuracy of this interpretation was done yet, a very high relative permeability is observed for the water phase at the end point (k<sub>rw</sub> at S<sub>orw</sub> is around 0.6), which confirms the intermediate wettability given by the USBM index (-0.07). These results are also very consistent with relative permeability curves derived from displacement experiments on the same rock type.

### **Numerical Simulations**

#### Principle

The above results were obtained by simple analytical calculations using the experimental results recorded after stabilization of both differential pressure and saturation profile. As the transient evolution of the differential pressure was recorded as a function of time, it is also possible to simulate numerically the whole experiment in order to derive continuous relative permeability curves. We have used the IFP reservoir code adapted to laboratory conditions (ATHOS – Implicit simulations were performed after a sensitivity study on the number of grid cells) to run these simulations. The main specificity of the semi-dynamic experiment concerns the outlet boundary condition, as the non injecting phase continuously washes this face. In the simulation, this condition was handled using an

artificial cell of infinite porosity (numerical way to have an infinite volume), very high permeability and zero capillary pressure. This cell enables to maintain a zero capillary pressure at the outlet and allows the spontaneous imbibition of the non injected phase (the saturation of this phase in this cell remains very close to unity throughout the simulation, due to the infinite porosity). All the simulations were performed in 1D using 0.5-cm grid cells.

The simulations were performed using the capillary pressure curves derived from the analytical calculations (previous part) and the  $k_r$ -curves were adjusted until a good match of the transient evolution of the differential pressure was obtained. The  $k_r$ -points derived from the analytical calculations were used as a starting point for the injecting phase.

### Results

The history matching of the saturation profiles and the differential pressure is plotted in Figures 9 and 10. A good agreement was reached during all the steps of the forced imbibition process. The inlet water saturation values at the different water injection rates are particularly well reproduced as well as the level of the stabilized pressure drop along the core. The difference between simulated and experimental saturations at the outlet may be due to a non-uniform initial saturation profile ( $S_{wi}$ -profile in Figure 5), to heterogeneity or to wettability artefacts (Spinler et al., [11; 12]).

The corresponding set of adjusted  $k_r$ -curves is given in Figure 11. From the local saturation measurements, the  $k_r$ -curves are representative for water saturation higher than 0.5. As expected, the  $k_{rw}$ -curve is very consistent with the points derived from the analytical calculation. The  $k_{ro}$ -curve enables to explore the asymptotic behavior of the oil phase at the low residual saturation. This curve is representative even at low residual oil saturation because the semi-dynamic method relies on a balance between viscous and capillary forces. When a conventional displacement imbibition experiment can only detect  $k_{ro}$  values of the order of  $10^{-3} - 10^{-4}$  because of the limited volume of water that can be injected at high flow rate, the semi-dynamic method enables to capture the whole behavior down to  $k_{ro}$  of the order of  $10^{-6}$  because about 65 PV were injected at the high injection rate step (500 cm<sup>3</sup>/hr).

However, we recall that the  $k_{ro}$  curve is determined by history matching and not by direct measurement. The accuracy in the determination of the low  $k_{ro}$  values near residual saturation has not been examined.

## **GUIDELINES FOR EXPERIMENT DURATION AND SATURATION ACCURACY**

The duration of the experiments strongly depends on permeability and on the length of the sample. This chapter gives some guidelines concerning such experiments and discusses the accuracy of the measurements.

### Scaling Laws for the Semi-Dynamic Method

Let us assume that we want to determine a given point of the capillary pressure curve with samples of different sizes. The semi-dynamic method is based on the balance between capillary and viscous forces. Consequently, if the length of the core or other properties are changed, the capillary number that represents the balance between the total pressure drop for the injected fluid across the sample ( $\Delta P$ ) and the capillary forces has to be kept constant. The capillary forces are generally assumed to scale as  $K^{-1/2}$  (for constant porosity  $\phi$ ).

Let us define the reference time,  $t_{ref}$ , as the time necessary to displace one pore volume :

$$t_{ref} = AL\phi / Q \quad (3)$$

where A is the cross section area, L the length of the sample and Q the volume flow rate.

At a given saturation, and for given relative permeabilities, the flow rate Q is proportional to the pressure gradient  $\Delta P/L$  (Darcy's law). Now assuming that  $\Delta P$  scales as the capillary pressure, e. g. as  $K^{-1/2}$  leads to:

$$Q \propto K\Delta P / L \propto K^{1/2} / L \quad (4)$$

Finally, the reference time  $t_{ref}$  scales as:

$$t_{ref} \propto L^2 K^{-1/2} \quad (5)$$

This scaling law has been validated numerically. Consequently, for a fixed permeability, the determination of a point of Pc- or kr-curves would be 16 times faster when a 32-cm sample is replaced by an 8-cm sample .

### Duration of a Complete Cycle

To estimate the total duration of a complete cycle for a 32cm sample ( $K=10\text{mD}$ ), two types of capillary pressure curves are considered (negative imbibitions and positive drainages derived from centrifuge measurements, and interpolations for the rest of the cycle)

- Type 1 without positive imbibition (Figure 12a),
- Type 2 with a slight positive imbibition (Figure 12b).

For both cases, the same kr-curves were used.

The procedure is similar to the one used for actual experiments:

- 3 steps are used when small saturation variations are expected (40, 5 and 0 cm<sup>3</sup>/h for positive imbibition and 5, 2 and 0 cm<sup>3</sup>/h for negative drainage),
- 6 steps are used where large saturation variations are expected (0.25, 0.5, 1, 2, 5 and 10 cm<sup>3</sup>/h for negative imbibition and 0.25, 0.5, 1, 5, 10 and 20 cm<sup>3</sup>/h for positive drainage),
- a step is changed when production is stable during 2 days.



For the Pc-curve of Type 2 (slight positive imbibition), the total duration is around 200 days (Figure 13). For the Pc-curve of Type 1 (no positive imbibition), the total duration of the cycle is of the order of 110 days.

Consequently, the duration of a Pc-cycle is not only linked to permeability and length of the sample, it is strongly increased when spontaneous displacements can occur.

### **Accuracy of the Measurement**

The only limit on the size of the core sample is the accuracy on the measurements. With our experimental setup, there are mainly two sources of limitations: accuracy in saturation measurement and flow perturbations due to the end pieces.

#### Accuracy in Saturation Measurements

The X ray beam is an 8-mm diameter cylinder. Each measurement requires about 1 minute. With the semi-dynamic method, the Pc-curve is determined only at the inlet of the sample. Consequently, only the local saturation at the inlet is needed. Nevertheless, average saturation is also used to check and recalibrate this local saturation. In addition, the saturation profile can also be useful for the validation of experiments by numerical simulations of the displacement.

We have studied the accuracy in determining the average saturation over the total length of the sample when the number of points of measurement is increased. We have chosen a representative saturation profile obtained from numerical simulations (Figure 14) and calculated the saturation measured by an 8-mm diameter beam. Figure 15 shows that the true average saturation is obtained with 18-20 points of measurement. There is no difficulty making such a measurement with a spacing of 4 mm.

The simulation also shows that the inlet saturation is always measured with a good accuracy, due to the flat shape of the saturation profile near the inlet when equilibrium is reached.

#### Effect of the End Pieces

The fluids are injected through end pieces with spiral shaped grooves. There is a perturbation of the flow at the inlet and outlet due to the presence of solid parts and grooves (the velocity is not exactly parallel close to the end faces). For flow in porous media, the perturbation affects a distance equal to the transverse dimension of the obstacle (Laplacian field). The distance between the grooves is equal to 1 mm and consequently, the perturbed zone extends over 1 mm, which is of the order of 1% of the total length of the sample. We can estimate the error due to this effect to be less than 1% for an 80-mm sample.

#### Conclusion

The measurements can be performed on 8-cm long samples with an accuracy comparable to the one obtained on the 32-cm long sample, but with the advantage of being 16 times faster.

## CONCLUSIONS

A prototype, based on the semi-dynamic method, was developed in order to measure the whole Pc-cycle of core samples at full reservoir conditions. The Pc-curves allow to determine the USBM wettability index, under pressure and temperature. Parallel to the Pc-measurements, the relative permeability can be obtained through analytical interpretation or through simulations of the transient data (history matching). The first experiment on a carbonate sample gives consistent results.

The duration of such experiments varies with the square length and the inverse of the square root of permeability of the sample. The longest step is the positive imbibition. If a sample with a given length is considered, for a better accuracy, there is a minimum number of local saturation points to be investigated.

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## NOMENCLATURE

A	cross-section area of the sample	$\langle S \rangle$	average saturation
K	single-phase permeability	Swi	irreducible water saturation
Kr	relative permeability	t <sub>ref</sub>	reference time
L	length of the sample	$\phi$	Porosity
N	X-ray profile (photons/s)	$\mu$	viscosity
Pc	capillary pressure	$\Delta P$	differential pressure
Pi	inlet pressure	indices o,w:	oil, water
q	volume flow rate	indice i:	inlet
S	saturation		

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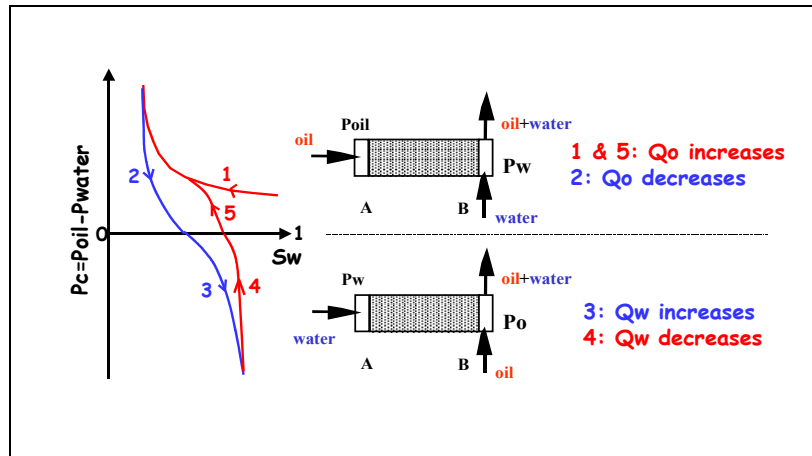


Figure 1: Capillary pressure curves definition - Principle of the semi-dynamic method

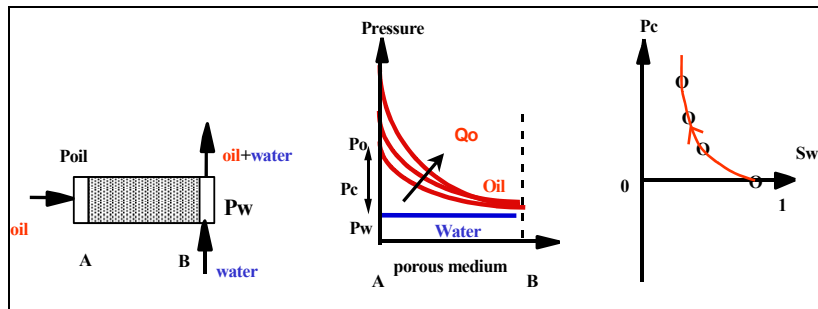


Figure 2: Semi-dynamic method: positive drainage

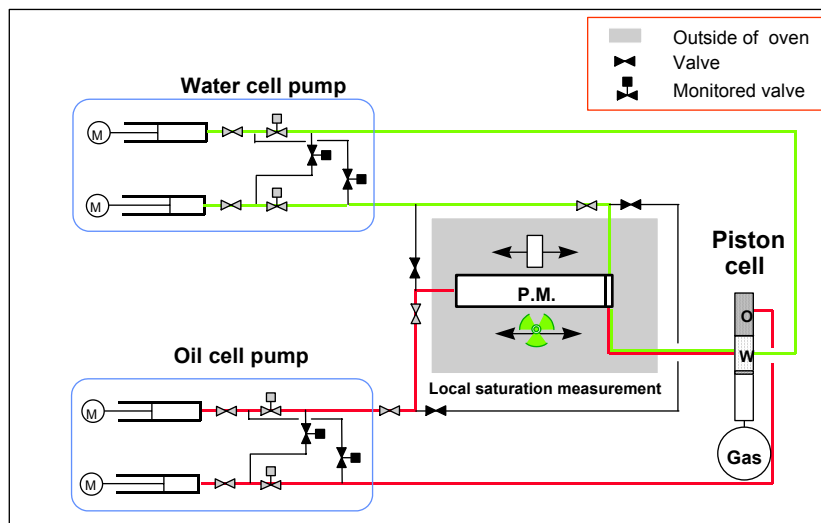
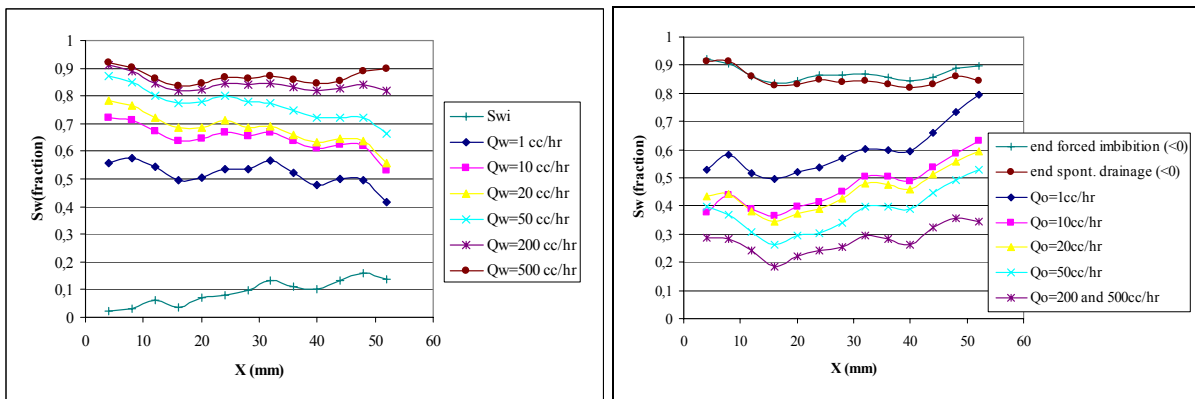


Figure 3: Principle of the semi-dynamic equipment



Figure 4: Semi-dynamic device



(a)

(b)

Figure 5: Saturation profiles during imbibition (a) and drainage (b)

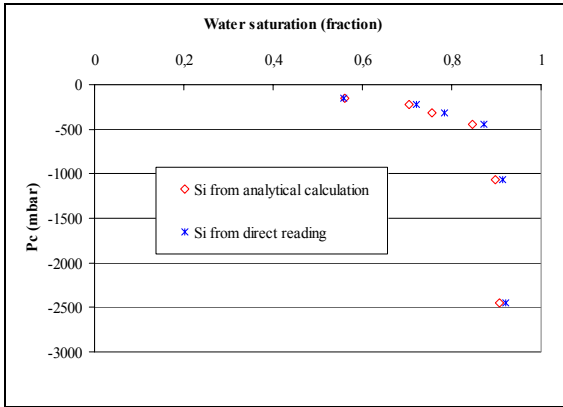


Figure 6: Influence of the inlet saturation on the Pc-curve

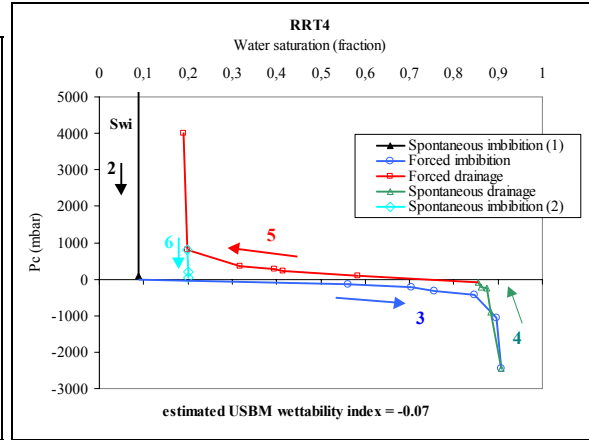


Figure 7: Pc-cycle and USBM Index

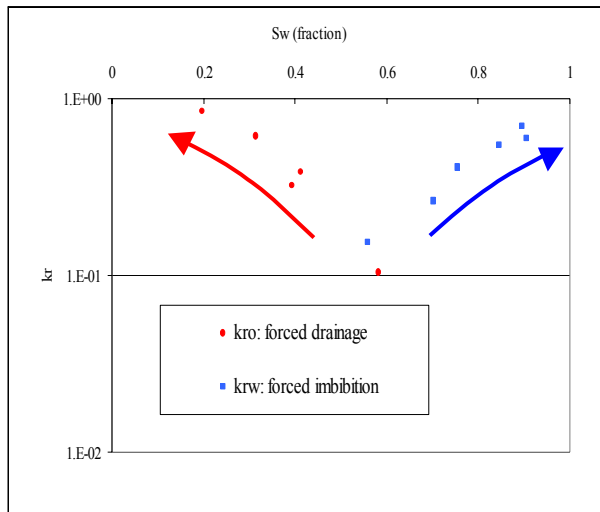


Figure 8: kr-points of the injecting phase derived from analytical calculation

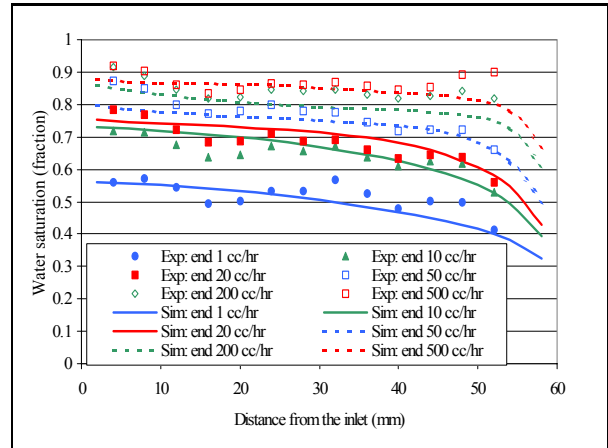


Figure 9: Simulation of saturation profiles as a function of time

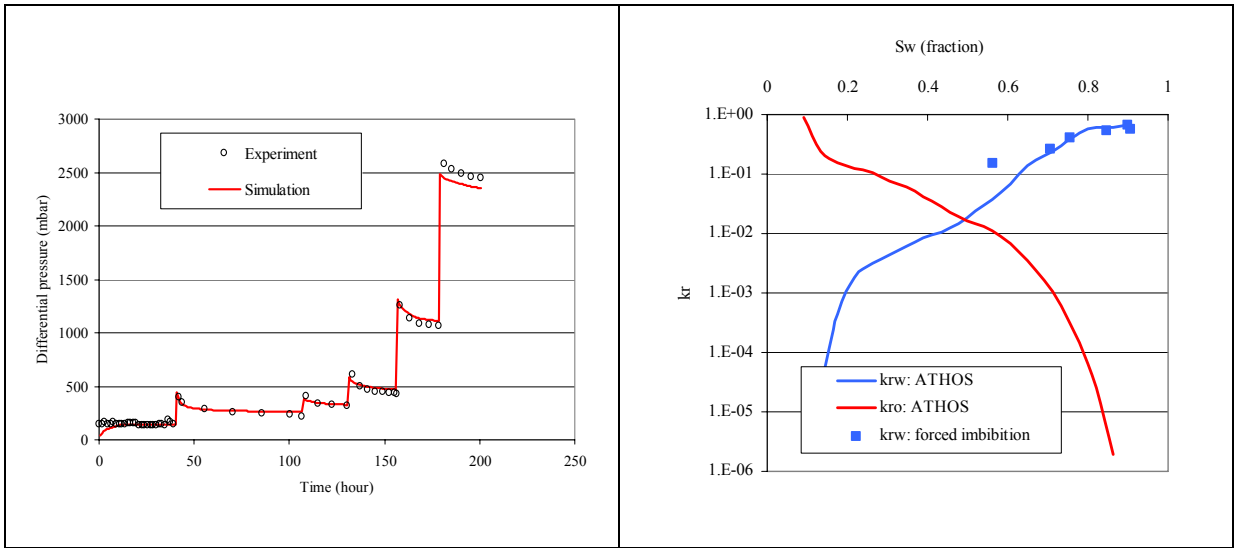
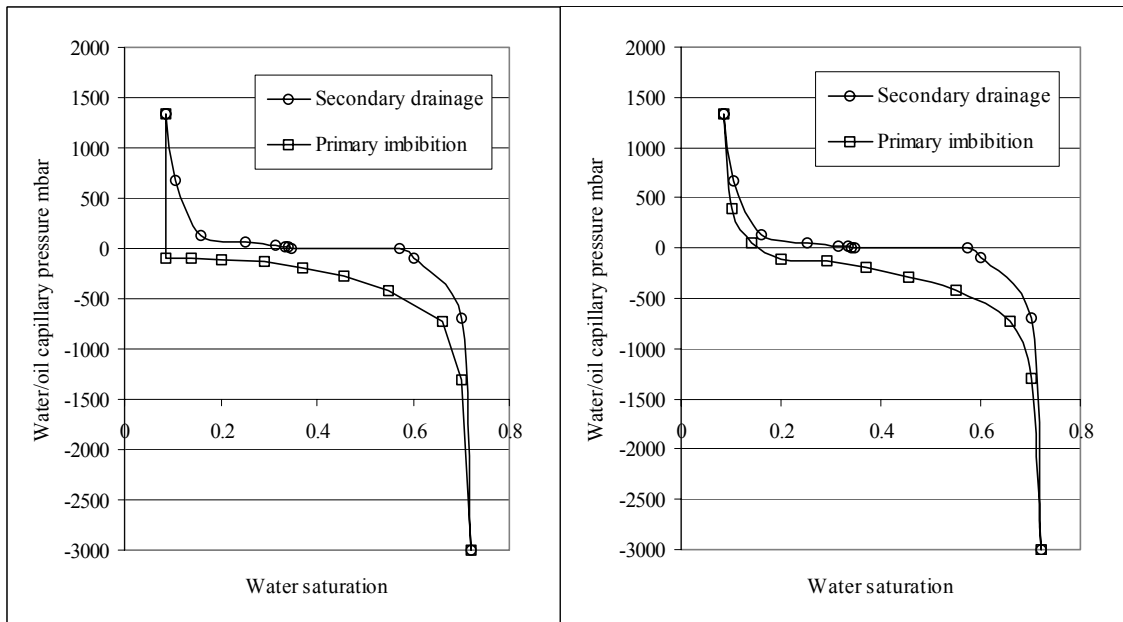


Figure 10: Simulation of  $\Delta P$  as a function of time

Figure 11: oil/water  $k_r$ -curves obtained by history matching



(a)

(b)

Figure 12: Capillary pressure curves used in the simulations

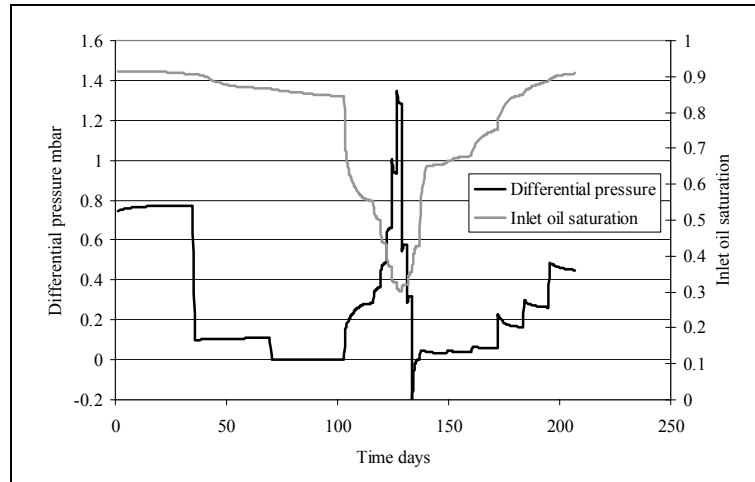


Figure 13: Total duration for the Pc-curve with positive imbibition

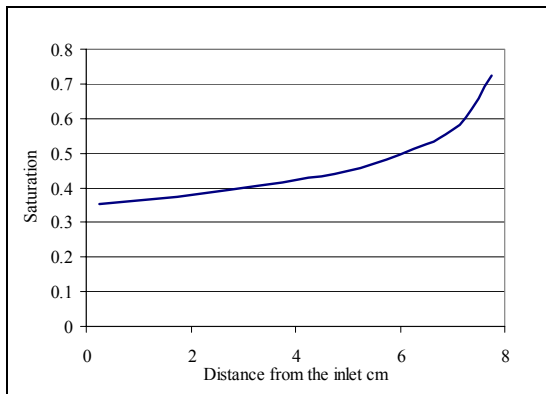


Figure 14: Saturation profile and accuracy

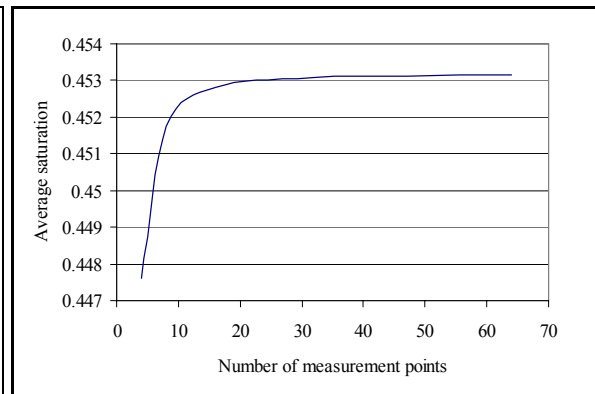


Figure 15: Accuracy on the average saturation determined with local measurement